Quantitation and Ratio Determination of Uranium Isotopes in Water and Soil Using Inductively Coupled Plasma Mass Spectrometry (ICP-MS)

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Introduction

- Uranium – overview
- Sample prep:
  - water – EPA 3020
  - Soils – EPA 3052 (modified)
- Analysis – ICP-MS
  - water – EPA 200.8
  - soils – EPA 6020
  - Quality Controls
- ICP-MS and α-Spec
- Summary
- Questions

Introduction

- Ubiquitous element
- Naturally Occurring U Isotopes:

  234 (0.0055%, 0.245 E6 yr.), 235 (0.72%, 703 E6 yr.), 238 (99.275%, 4,468 E6 yr.)
- Natural U235/238 atomic Ratio: 7.2 x 10-3
- Natural U234/238 α activity Ratio: 1 (secular equilibrium)
- Used for fuel in atomic energy and warfare
- Depleted Uranium DU: 235 Isotope Quantity Reduced

  U235/238 atomic Ratio: 2 x 10-3

Sample Preparation

- **Water - EPA 3020**
  - **Acid digestion**
- **Soils – EPA 3052 (modified)**
  - **Acidic microwave digestion**
  - **Complete digestion**
- **Ratio – Depends on Matrix (see methods above)**

Sample Analysis ICP-MS

- Water - EPA 6020
- Soils – EPA 200.8
- Ratios – In house Method

Sample Analysis QC

- Sample Duplicates – precision check
- Blanks – contamination check
- Laboratory Control Samples – accuracy check
- Matrix Spikes – matrix effect
- *Mass bias correction standard

Analysis Recovery QC

	Water	Soil	Ratio
Duplicates	---	≤ 20 % RPD	---
Blanks	< RL	< RL	---
LCS	± 15 %	± 20 %	---
MS	± 30 %	± 30 %	---
Inst Spike	± 20 %	± 20 %	---
ISA / ISB	---	± 20 %	---

Common Analysis Techniques

- α Spectroscopy
- ICP-MS

α Spectroscopy

- Measures 234 and 238 isotopes
  U-234 from the Uranium Decay Series
  U238 → Th234 → Pa234 → U234 → Th230 → …..
- Sample preparation required (matrix removed)
- Tracer added for quantification
- Measure α particles from radioactive decay
- Ratio and Concentration in same analysis
- Detection limits – depends on count time

ICP- MS

- Measures 235 and 238 isotope ions
  - 235 from the Actinium Decay Series
  - U235 → Th231 → Pa231 → Ac227 → …..
- Sample preparation
- Count ions
- Conc. and Ratio: two different analyses
- Detection limit: matrix and instrument

ICP- MS 235/238 Ratio Comparison

Uncorrected Bias	Corrected Bias
6.51 x10-3	7.24 x10-3
6.82 x10-3	7.18 x10-3
6.58 x10-3	7.25 x10-3
6.24 x10-3	7.14 x10-3
6.74 x10-3	7.22 x10-3

Accepted Ratio value 7.26 x10-3

ICP- MS

- Measures 235 and 238 isotope ions
  - 235 from the Actinium Decay Series
  - U235 → Th231 → Pa231 → Ac227 → …..
- Sample preparation
- Count ions
- Conc. and Ratio: two different analyses
- Detection limit: matrix and instrument

Choices

- α Spectroscopy
- ICP-MS

- Questions you need answered:
  - Concentration?
  - Ratio?

ICP- MS and α Spec

ICP-MS	α Spec
235 Conc. Sufficient for ratio	Long count time
Ratio & Conc. Separate Analysis	Ratio & Conc. Same Analysis
Correct ratio? – bias, conc.	Correct ratio – recoil effect
100 mL sample	1 L sample

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Questions

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